

PATENT ABSTRACTS OF JAPAN

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(54) YTTRIUM-ALUMINUM-GARNET CALCINED POWDER AND PRODUCTION OF YTTRIUM-ALUMINUM-GARNET SINTERED COMPACT USING THE SAME

(57)Abstract:

PURPOSE: To produce a sintered compact having satisfactory productivity and translucency and free from voids and defects in an arbitrary shape at a low cost with high productivity without adversely affecting the environment by mixing Al₂O₃ powder with Y₂O₃ powder, calcining the resultant mixture and specifying ratio in X-ray diffraction intensity.

CONSTITUTION: Powdery Al₂O₃ and powdery Y₂O₃ each having $\geq 99.0\%$ purity $\geq 5\text{m}^2/\text{g}$ BET specific surface area and $\leq 1\mu\text{m}$ particle diameter are mixed so that the ratio of Al₂O₃:Y₂O₃ is regulated to 0.43:0.57 and the resultant mixture is calcined at 1,000-1,600°C for $\geq 0.5\text{hr}$ until $\geq 50\%$ rate of formation of YAG is attained. The calcined mixture is pulverized to prepare powdery starting material, a solvent is added and they are

mixed and pulverized with a pot mill, etc., to obtain calcined particles having $\leq 2\mu\text{m}$ particle diameter. These particles are dried, screened and press-compacted to $\geq 1\text{g}/\text{cm}^3$ density so as to minimize voids in a formed sintered compact. The resultant compact is heated to 1,600-1,900°C max. temp. at 50-300°C/hr rate of temp. rise at $\leq 10\text{Torr}$ degree of vacuum and it is held at the max. temp. for 2-20hr to obtain the objective YAG sintered compact having a ratio in X-ray diffraction intensity satisfying the formula and contg. $\geq 60\%$ YAG having 30μm average grain diameter.

CLAIMS

[Claim(s)]

[Claim 1] Purity is 99.0% or more of aluminum 2O3, respectively. Powder and Y2 O3 Yttrium aluminum garnet temporary-quenching powder characterized by coming to carry out temporary quenching at 1000-1600 degrees C, and an X diffraction intensity ratio filling YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y2O3(222)+aluminum 2O3 (113)>=0.5 after mixing powder.

[Claim 2] The manufacture approach of the yttrium aluminum garnet sintered compact characterized by calcinating at the temperature of 1600-1900 degrees C among a vacuum or a reducing atmosphere after fabricating yttrium aluminum garnet powder according to claim 1 in a desired configuration.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the YAG powder used for the manufacture approach of a translucency polycrystal yttrium aluminum garnet (it is called Following YAG) sintered compact, and this.

[0002]

[Description of the Prior Art] As for YAG (Y3 aluminum 5O12), the attempt which obtains a translucency sintered compact by various kinds of manufacture approaches since it is good as the transparent body is made that grain boundary dispersion of light cannot take place easily since a crystal mold is a cubic.

[0003] As the concrete manufacture approach, it is the approach and aluminum 2O3 which are created with a single crystal. Powder and Y2 O3 The raw material obtained in powder with HIP processing, the approach of carrying out hotpress baking, or the urea precipitation method of yttrium ion and aluminum ion is fabricated, and it is manufactured by the approach of calcinating etc. (for example, refer to JP,54-8369,B).

[0004] Moreover, YAG obtained by the above-mentioned manufacture approach is used for the aperture material for clocks, the arc tube for electric-discharge lamps, etc. using the descriptions, such as translucency and corrosion resistance.

[0005]

[Problem(s) to be Solved by the Invention] However, by single crystal composition, it is expensive and there was a problem that it was difficult to manufacture in the configuration of arbitration among the manufacture approaches of Above YAG.

Moreover, when based on HIP processing, equipment became large, and there was a problem that productivity was not good. Furthermore, when manufacturing with a hotpress, carbon went into the sintered compact from the carbon used for a die, and there was a fault that transparency fell.

[0006] Moreover, in a urea precipitation method, an ammonia steam needs to be processed and there was a possibility of having a bad influence on an environment.

[0007] Furthermore, these people are aluminum 2O3. Powder and Y2 O3 After mixing powder, temporary quenching was carried out, it considered as raw material powder, and the manufacture approach of fabricating and calcinating this raw material powder in a predetermined configuration is proposed (refer to JP,6-107456,A). However, in this manufacture approach, since the yield of the YAG crystal in the

powder after temporary quenching was about 10 - 50%, in connection with the cubical expansion at the time of changing from YAM to YAG at the time of this baking, it was easy to produce a void and a defect in the sintered compact, and it had the problem of being hard to obtain the high YAG sintered compact of translucency.

[0008]

[Means for Solving the Problem] Then, for this invention, purity is 99.0% or more of aluminum 2O₃, respectively. Powder and Y₂ O₃ After mixing powder, It comes to carry out temporary quenching at 1000-1600 degrees C. An X diffraction intensity ratio It is characterized by the yttrium aluminum garnet temporary-quenching powder which fills YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y₂O₃(222)+aluminum 2O₃ (113)>=0.5.

[0009] Moreover, after this invention fabricates the above-mentioned yttrium aluminum garnet temporary-quenching powder in a predetermined configuration, it is characterized by calcinating at the temperature of 1600-1900 degrees C, and manufacturing an yttrium aluminum garnet sintered compact.

[0010] Here, purity is 99.0% or more of aluminum 2O₃, respectively. Powder and Y₂ O₃ Powder was used for an impurity existing and the translucency falling into a sintered compact, when purity was lower than 99.0%.

[0011] Moreover, aluminum 2O₃ Powder and Y₂ O₃ It is aluminum 2O₃ by this temporary quenching to carry out temporary quenching of the powder at 1000-1600 degrees C. Y₂ O₃ While making it combine, generating YAG and controlling the void at the time of this baking, and generating of a defect, it is for holding activation. At this time, temporary-quenching temperature was made into 1000-1600 degrees C, because activity will become low if temporary-quenching temperature is higher than 1600 degrees C, baking in an elevated temperature was [if lower than 1000 degrees C, the yield of a YAG crystal will become 50% or less, / it is to become easy to generate a void and a defect in the sintering process in this baking, and] needed on the other hand and the grindability after temporary quenching also worsened.

[0012] Furthermore, it is aluminum 2O₃ by the above-mentioned temporary quenching. Y₂ O₃ Although it changes from YAM from mixed powder to YAP to YAG, respectively, to YAM from YAP In this invention, each peak intensity ratio when an X diffraction analyzes the obtained temporary-quenching powder found out that what is necessary was just to fill YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y₂O₃(222)+aluminum 2O₃ (113)>=0.5. This formula expresses the yield of YAG in temporary-quenching powder, and it means that a YAG yield is 50% or more as the above-mentioned X diffraction intensity ratio is 0.5 or more. It is because it becomes difficult to become easy to produce a void and a defect by the cubical expansion at the time of these intermediate fields changing to YAG in the sintering process in this baking, and to make translucency of a sintered compact high, since, as for having limited the above-mentioned X diffraction intensity ratio, an X diffraction intensity ratio contains many intermediate fields, such as aluminum 2O₃, YAP, and YAM, as less than 0.5, i.e., a YAG yield, are less than 50% here.

[0013] In addition, translucency of a sintered compact can be made higher by making the above-mentioned temporary-quenching temperature into the range of 1200-1350 degrees C preferably, and making a YAG yield into 70 - 85% of range.

[0014] Next, after fabricating the obtained temporary-quenching powder in a predetermined configuration, if lower than 1600 degrees C, it will calcinate at 1600-1900 degrees C for sintering being inadequate, and not carrying out eburnation, but translucency making it low, and when higher than 1900 degrees C, it is because abnormality grain growth arises, it incorporates pore in a grain, evaporation of YAG

arises not only translucency falls, but and it becomes impossible to create a homogeneous sintered compact.

[0015] Moreover, this baking is performed in a vacuum or a reducing atmosphere, because the eburnation of a sintered compact can be attained easily.

[0016] For the YAG sintered compact of this invention, purity is aluminum 2O3 5m² of BET specific surface areas / more than 99.0% or more and g, respectively, for example. Powder and Y2 O3 About powder, it is aluminum 2O3. : Temporary quenching is carried out after it adjusts so that Y2 O3 may be set to 0.43:0.57, and mixing until the above-mentioned YAG yield becomes 50% or more at 1000-1600 degrees C for 0.5 hours or more. In addition, aluminum 2O3 Powder and Y2 O3 In order to prevent abnormality grain growth of YAG, as for a powdered particle size, it is desirable that it is 2 micrometers or less, respectively.

[0017] And this is ground and it considers as raw material powder, and a predetermined solvent is added in this raw material, and preferential grinding of this is carried out to it by the pot mill, a tumbling mill, etc. 2 micrometers or less of particles of temporary-quenching powder are preferably set to 1 micrometer or less. Then, after drying this, a particle size regulation is carried out with 80-mesh pass. This is fabricated in the configuration of arbitration by the predetermined shaping means, for example, the die press, the cold isostatic press (CIP), extrusion molding, etc. For example, when based on the die press, it is 2.5 ton/cm². It pressurizes by the above pressure and the consistency of a generation form is made as high as possible. As for the consistency of a Plastic solid, it is desirable to consider as three or more 2.1 g/cm in order to control the void of a sintered compact to the minimum.

[0018] And this baking carries out by a degree of vacuum holding [in / preferably / 1x10 to 2 or less torrs / the vacuum ambient atmosphere of 1x10 to 3 or less torrs] a programming rate at 1600-1900 degrees C of maximum temperatures for 2 to 20 hours by making desirable 50-300 degrees C /in 50-100 degrees C/hour an hour. In addition, you may calcinate instead of a vacuum ambient atmosphere by the reducing atmosphere of a hydrogen ambient atmosphere or nitrogen-gas-atmosphere mind.

[0019] According to the manufacture approach of such this invention, in order that a YAG yield may use 50% or more of temporary-quenching powder, generating of the void and defect accompanying the volume change from YAM to YAG at the time of this baking is controlled, and it becomes possible to produce a uniform sintered compact. Consequently, the obtained YAG sintered compact contains YAG of 30 micrometers or less of diameters of average crystal grain 60% or more, and the straight-line permeability of the light per thickness of 1mm can make it 80% or more suitably 70% or more.

[0020] Moreover, since it is not necessary to use baking of the raw material by single crystal composition, HIP processing, the hotpress, and the urea precipitation method etc. like before and manufactures by general ordinary pressure baking according to this invention, a translucency YAG sintered compact can be obtained cheaply and easily. Furthermore, since the transparent body is manufactured using a polycrystal YAG sintered compact, it can become a low price, reinforcement can be stabilized, the configuration of arbitration can be manufactured easily, and polishing etc. can be processed easily.

[0021] Furthermore, since translucency, corrosion resistance, and abrasion resistance are excellent, this YAG sintered compact can be used suitable for various applications, such as the window part material for clocks, the arc tube for electric-discharge lamps, a member for optical connecters, various smooth substrates for electronic parts, a substrate for magnetic disks, a member for an ornament, and seal.

[0022]

[Example] aluminum 2O₃ whose purity is 99.7%, 5m² of BET specific surface areas / g, and the mean particle diameter of 1 micrometer as a start raw material first, respectively 129g of powder, and Y₂O₃ 171g of powder was fed into the poly pot with high grade alumina-balls 600g and IPA300g as a solvent, and it carried out preferential grinding by the tumbling mill for 24 hours. After carrying out through desiccation of the obtained mixture at 325 meshes, uniform powder was obtained through 80 meshes. Temporary quenching of this powder was carried out at various temperature at the atmospheric-air furnace, and the peak intensity of YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y₂O₃(222)+aluminum 2O₃ (113) was measured for the obtained temporary-quenching powder according to the X diffraction.

[0023] Next, this temporary-quenching powder was again fed into the poly pot with high grade alumina-balls 600g and IPA300g of a solvent, and preferential grinding was carried out by the tumbling mill for 24 hours. After making 325 meshes carry out through desiccation of the ground powder, through and uniform powder were obtained to 80 meshes. The raw consistency produced the Plastic solid of 2.5m³ / g for this powder using a 1 shaft press and CIP.

[0024] After calcinating on the conditions which show this Plastic solid in Table 1 and polishing the obtained sintered compact in thickness of 1mm, diamond paste with a particle size of 1 micrometer performed mirror plane finishing. Straight-line permeability with a wavelength of 600nm was measured to this sintered compact.

[0025] A result is as being shown in Table 1. It turned out that temporary quenching is not performed, or that (14 No.1, 15) whose YAG yield the X diffraction intensity ratio after temporary quenching was less than 0.5, and was less than 50% has the straight-line permeability of a sintered compact as lower as less than 60% than this result, and translucency is bad. On the other hand, since No.8 had temporary-quenching temperature higher than 1600 degrees C, although the X diffraction intensity ratio was 1, the activity of temporary-quenching powder became low and the straight-line permeability of a sintered compact was as low [the intensity ratio] as less than 70%.

[0026] As for each thing (No.2-7, 9-13) which made temporary-quenching temperature 1000-1600 degrees C, and made the YAG yield after 0.5 or more, i.e., temporary quenching, 50% or more for the X diffraction intensity ratio, the straight-line permeability of a sintered compact indicated high translucency to be 70% or more to these. Straight-line permeability became 80% or more, and what made temporary-quenching temperature 1200-1350 degrees C, made the X diffraction intensity ratio to 0.7 to 0.85, made the YAG yield 70 - 85%, and calcinated this temporary-quenching powder especially for 10 hours or more at the programming rate of 50-100 degrees C/hour and 1800-1900 degrees C (No.10-12) showed high translucency especially.

[0027]

[Table 1]

[0028] Moreover, the example of a chart Fig. by the X diffraction of the above-mentioned temporary-quenching powder is shown in drawing 1 . The YAG yield as which most peaks of YAG are not regarded as shown in drawing 1 (A), but temporary-quenching temperature is defined by the above-mentioned X diffraction intensity ratio in the example of a comparison 1000 degrees C or less was less than 50%. On the other hand, in this invention example which made temporary-quenching

temperature 1000-1600 degrees C, many peaks of YAG were detected and the YAG yield was 50% or more.

[0029] Furthermore, the temperature when carrying out actual baking using these temporary-quenching powder and the relation of an elongation percentage (contraction) are shown in drawing 2. When it calcinates using the temporary-quenching powder of the example of a comparison whose YAG yield is less than 50% and the arrow head in the middle of burning shrinkage shows as shown in drawing 2 (A), it turns out that the volume is expanding conversely. This shows that cubical expansion breaks out, in case YAM changes to YAG, as mentioned above, and it will make translucency low that it is easy to produce a void and a defect in a sintered compact in connection with this cubical expansion.

[0030] On the other hand, when the temporary-quenching powder of this invention example whose YAG yield is 50% or more is used, as shown in drawing 2 (B), it turns out that cubical expansion does not happen in the middle of burning shrinkage, therefore the high sintered compact of translucency is obtained.

[0031] In addition, at the above-mentioned example, it is aluminum 2O3 of 99.7% of purity as a start raw material. Powder and Y2 O3 Although powder was used, when it was 99.0% or more, about the purity of these powder, it checked that there was the same effectiveness as the above-mentioned example.

[0032]

[Effect of the Invention] Thus, according to this invention, purity is 99.0% or more of aluminum 2O3, respectively. Powder and Y2 O3 After mixing powder, It is characterized by the yttrium aluminum garnet powder with which it comes to carry out temporary quenching at 1000-1600 degrees C, and an X diffraction intensity ratio fills $YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y2O3(222)+aluminum\ 2O3(113)>=0.5$. By calcinating at the temperature of 1600-1900 degrees C, and having manufactured the yttrium aluminum garnet sintered compact, after fabricating this yttrium aluminum garnet powder in a predetermined configuration Generating of the void and defect accompanying cubical expansion by the change to YAG at the time of this baking from YAM can be controlled, and a uniform sintered compact can be obtained. Consequently, the high YAG sintered compact of translucency [as / whose straight-line permeability of the light field in the thickness of 1mm is 70% or more] can be manufactured cheaply and easily. Moreover, since this YAG sintered compact is the polycrystalline substance, its reinforcement can be stable, can manufacture the configuration of arbitration easily and can process polishing etc. easily, it can be used suitable for various applications.

TECHNICAL FIELD

[Industrial Application] This invention relates to the YAG powder used for the manufacture approach of a translucency polycrystal yttrium aluminum garnet (it is called Following YAG) sintered compact, and this.

PRIOR ART

[Description of the Prior Art] As for YAG (Y3 aluminum 5O12), the attempt which obtains a translucency sintered compact by various kinds of manufacture approaches since it is good as the transparent body is made that grain boundary dispersion of light

cannot take place easily since a crystal mold is a cubic.

[0003] As the concrete manufacture approach, it is the approach and aluminum 2O₃ which are created with a single crystal. Powder and Y₂ O₃ The raw material obtained in powder with HIP processing, the approach of carrying out hotpress baking, or the urea precipitation method of yttrium ion and aluminum ion is fabricated, and it is manufactured by the approach of calcinating etc. (for example, refer to JP,54-8369,B). [0004] Moreover, YAG obtained by the above-mentioned manufacture approach is used for the aperture material for clocks, the arc tube for electric-discharge lamps, etc. using the descriptions, such as translucency and corrosion resistance.

EFFECT OF THE INVENTION

[Effect of the Invention] Thus, according to this invention, purity is 99.0% or more of aluminum 2O₃, respectively. Powder and Y₂ O₃ After mixing powder, It is characterized by the yttrium aluminum garnet powder with which it comes to carry out temporary quenching at 1000-1600 degrees C, and an X diffraction intensity ratio fills YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y2O3(222)+aluminum 2O₃ (113)>=0.5. By calcinating at the temperature of 1600-1900 degrees C, and having manufactured the yttrium aluminum garnet sintered compact, after fabricating this yttrium aluminum garnet powder in a predetermined configuration Generating of the void and defect accompanying cubical expansion by the change to YAG at the time of this baking from YAM can be controlled, and a uniform sintered compact can be obtained. Consequently, the high YAG sintered compact of translucency [as / whose straight-line permeability of the light field in the thickness of 1mm is 70% or more] can be manufactured cheaply and easily. Moreover, since this YAG sintered compact is the polycrystalline substance, its reinforcement can be stable, can manufacture the configuration of arbitration easily and can process polishing etc. easily, it can be used suitable for various applications.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] However, by single crystal composition, it is expensive and there was a problem that it was difficult to manufacture in the configuration of arbitration among the manufacture approaches of Above YAG. Moreover, when based on HIP processing, equipment became large, and there was a problem that productivity was not good. Furthermore, when manufacturing with a hotpress, carbon went into the sintered compact from the carbon used for a die, and there was a fault that transparency fell.

[0006] Moreover, in a urea precipitation method, an ammonia steam needs to be processed and there was a possibility of having a bad influence on an environment.

[0007] Furthermore, these people are aluminum 2O₃. Powder and Y₂ O₃ After mixing powder, temporary quenching was carried out, it considered as raw material powder, and the manufacture approach of fabricating and calcinating this raw material powder in a predetermined configuration is proposed (refer to JP,6-107456,A). However, in this manufacture approach, since the yield of the YAG crystal in the powder after temporary quenching was about 10 - 50%, in connection with the cubical expansion at the time of changing from YAM to YAG at the time of this baking, it

was easy to produce a void and a defect in the sintered compact, and it had the problem of being hard to obtain the high YAG sintered compact of translucency.

MEANS

[Means for Solving the Problem] Then, for this invention, purity is 99.0% or more of aluminum 2O3, respectively. Powder and Y2 O3 After mixing powder, It comes to carry out temporary quenching at 1000-1600 degrees C. An X diffraction intensity ratio It is characterized by the yttrium aluminum garnet temporary-quenching powder which fills $YAG(420)/YAG(420)+YAM(-122)+YAP(121)+Y2O3(222)+aluminum 2O3 (113)>=0.5$.

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[0013] In addition, translucency of a sintered compact can be made higher by making the above-mentioned temporary-quenching temperature into the range of 1200-1350 degrees C preferably, and making a YAG yield into 70 - 85% of range.

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EXAMPLE

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[0024] After calcinating on the conditions which show this Plastic solid in Table 1 and polishing the obtained sintered compact in thickness of 1mm, diamond paste with a particle size of 1 micrometer performed mirror plane finishing. Straight-line permeability with a wavelength of 600nm was measured to this sintered compact.

[0025] A result is as being shown in Table 1. It turned out that temporary quenching is not performed, or that (14 No.1, 15) whose YAG yield the X diffraction intensity ratio after temporary quenching was less than 0.5, and was less than 50% has the straight-line permeability of a sintered compact as lower as less than 60% than this result, and translucency is bad. On the other hand, since No.8 had temporary-quenching temperature higher than 1600 degrees C, although the X diffraction intensity ratio was 1, the activity of temporary-quenching powder became low and the straight-line permeability of a sintered compact was as low [the intensity ratio] as less than 70%.

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[0027]

[Table 1]

[0028] Moreover, the example of a chart Fig. by the X diffraction of the above-

mentioned temporary-quenching powder is shown in drawing 1. The YAG yield as which most peaks of YAG are not regarded as shown in drawing 1 (A), but temporary-quenching temperature is defined by the above-mentioned X diffraction intensity ratio in the example of a comparison 1000 degrees C or less was less than 50%. On the other hand, in this invention example which made temporary-quenching temperature 1000-1600 degrees C, many peaks of YAG were detected and the YAG yield was 50% or more.

[0029] Furthermore, the temperature when carrying out actual baking using these temporary-quenching powder and the relation of an elongation percentage (contraction) are shown in drawing 2. When it calcinates using the temporary-quenching powder of the example of a comparison whose YAG yield is less than 50% and the arrow head in the middle of burning shrinkage shows as shown in drawing 2 (A), it turns out that the volume is expanding conversely. This shows that cubical expansion breaks out, in case YAM changes to YAG, as mentioned above, and it will make translucency low that it is easy to produce a void and a defect in a sintered compact in connection with this cubical expansion.

[0030] On the other hand, when the temporary-quenching powder of this invention example whose YAG yield is 50% or more is used, as shown in drawing 2 (B), it turns out that cubical expansion does not happen in the middle of burning shrinkage, therefore the high sintered compact of translucency is obtained.

[0031] In addition, at the above-mentioned example, it is aluminum 2O₃ of 99.7% of purity as a start raw material. Powder and Y₂O₃ Although powder was used, when it was 99.0% or more, about the purity of these powder, it checked that there was the same effectiveness as the above-mentioned example.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is an X diffraction chart Fig. in the YAG temporary-quenching powder of this invention example and the example of a comparison.

[Drawing 2] It is the graph which shows the contraction behavior at the time of baking using the YAG temporary-quenching powder of this invention example and the example of a comparison.